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CONTENTS

To Head Warp Sizing Research 395

Chemical Week in Boston 395

The Natural Pigments of Silk, *A Textile Foundation Publication.* Werner Bergmann 397

A Study of Warp Size Distribution on Continuous Filament Bright Viscose Rayon. William E. Yelland 409

Abstracts 421

Advertisements

Henry L. Scott Co., Providence, R. I. 394

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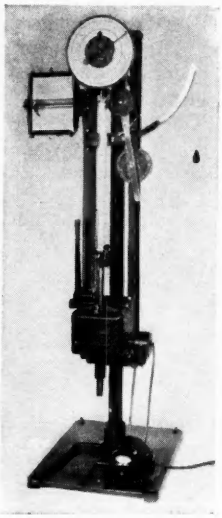

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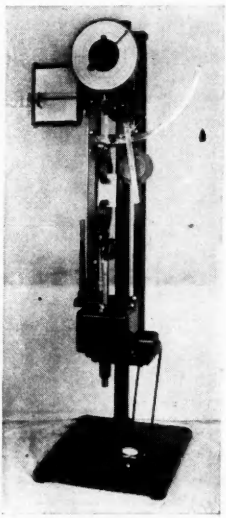

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TESTERS

To Head Warp Sizing Research

Carl R. Harris, manufacturing engineer, Erwin Cotton Mills Co., Durham, N. C., has accepted appointment as chairman of the Administrative Committee for the cotton and spun rayon warp sizing research to be conducted at North Carolina State Textile School, Raleigh, N. C., under the auspices of U. S. Institute for Textile Research, Inc. The other members of the committee, as previously reported, are Thomas Nelson, Dean of the School, and Dr. W. E. Yelland, who was director of the Institute's previous research on the sizing of filament viscose rayon, and is now with the research department of Corn Products Refining Co.

Mr. Harris was graduated from N. C. Textile School in 1917, has had a broad experience in cotton textile manufacturing since then, and was president of the Southern Textile Association in 1928–29.

The Tentative Program

The tentative program for the research is in part as follows:

"A practical study will be made of the sizing of spun rayon warps and of warps composed of mixtures of spun rayon and cotton. The work will be done at North Carolina State College Textile School. It will be limited at the start to a few representative size formulae. Warps will be slashed on commercial equipment, varying such conditions as the tension on the warp entering the size box, temperature of the size, pressure of the squeeze rolls, temperature of the drying cylinder, and moisture content of the yarn as it comes from the cylinder. The slasher will be equipped with instruments for measuring, recording, and controlling the level of the size and its temperature, the temperature of the surface of the drying cylinder, and the moisture content of the warp as it leaves the cylinder. Special attention will be given to the weaving of the warps, particularly with respect to the relative humidity and temperature of the air. To this end the looms will be located in a room in which humidity and temperature can be controlled. The distribution and penetration of the size, stiffness, flexibility, resistance to abrasion and performance in the loom of the sized yarns will be studied."

The complete program and information as to participation in this cooperatively financed research may be obtained by addressing the Secretary of U. S. Institute for Textile Research, Inc.

* * *

Chemical Week in Boston

WITH the semi-annual meeting of the American Chemical Society in Boston Sept. 11–15 (headquarters at the Hotel Statler), and the annual meeting of the American Association of Textile Chemists and Colorists scheduled for Sept. 15 and 16 at the Copley Plaza Hotel, that city will be the chemical centre of the country for at least one week.

This 19th annual meeting of the A. A. T. C. C. is held under the auspices of the Northern New England Section of which Clarence L. Nutting is chairman and John N. Dalton is secretary. The convention committee is as follows: Chairman, W. H. Zillesen; vice-chairman, J. A. Bryant; technical program, Carl Z. Draves; reservations and registration, Edward E. Bell; entertainment, M. J. Lane; reception, K. H. Barnard; publicity and exhibition, H. P. Selya; sightseeing, C. H. A. Schmitt. Council and research committee meetings are scheduled for Friday morning; the first technical session for that afternoon, this to be followed by a buffet supper and entertainment. The second technical session comes Saturday A. M., the afternoon being devoted to sightseeing and social activities, and the meeting closing with a banquet that evening.

A. A. T. C. C. Technical Papers

First Technical Session: The Dyeing of Nylon—A Preliminary Survey, by Philip H. Stott of E. I. du Pont de Nemours & Co.; Progress in Research from the Consumer Point of View, by Prof. Pauline Beery Mack of Penn. State College; Identification of the Newer Textile Fibres, by John H. Skinkle of Lowell Textile Institute; Semi-Resin Finishes for Cotton Piece Goods, by W. W. Trowell of Hercules Powder Co.; Flexibility and Drape as Measurable Properties of Fabric, by L. J. Winn, A. A. T. C. C. Research Assistant, and Prof. E. R. Schwarz of M. I. T.; Tintinoil Process, by Edward H. Schmidt of Borne Scrymser Co.; A New Type Water Repellant, by George A. Slowinske of E. I. du Pont de Nemours & Co.; Textone—A New Oxidizing Agent for the Treatment of Textiles, by G. P. Vincent, A. L. Dubeau and J. D. MacMahon of Mathieson Alkali Works.

Second Technical Session: Microscopic Observations of Wool Dyeing, by H. E. Millson, G. L. Royer and G. E. Wissemann of Calco Chemical Co.; A Motion Picture Study of Some Dyeing Phenomena, by R. H. Kienle and G. L. Royer of Calco Chemical Co.; Sodium Sulfate Error in Measuring the Acid Content of Carbonizing Baths, by H. C. Olsen and W. B. Prescott with Dr. H. C. Chapin of Lowell Textile Institute; New Concepts in the Chemistry of Wool, by Dr. Milton Harris, Director of Research for The Textile Foundation at the National Bureau of Standards; The Present Status of Casein Fibre, by E. O. Whittier and S. P. Gould of the Bureau of Dairy Industry; Surface Cation Active Compounds in the Textile Industry, by Jacob Katz of Warwick Chemical Co.; The Development of Methods for the Determination of the Causes of Damage Appearing in Wool After Dyeing, by Bertil A. Ryberg, Research Associate of the A. A. T. C. C. supported by the Textile Foundation at Lowell Textile Institute; Modern Dyeing of Staple and Spun Viscose Rayon, by Charles B. Ordway of American Aniline Products.

A. C. S. Textile Papers

At this writing the complete program for the American Chemical Society sessions is not available, but among the papers to be presented at the sessions of the Division of Cellulose Chemistry are several of particular interest to textile men; among these are the following: Wednesday A. M.—

(*Continued on page 417.*)

The Natural Pigments of Silk

By WERNER BERGMANN *

A Textile Foundation Publication

Introduction

MANY of the different varieties of silkworms of commercial importance produce more or less intensely colored cocoons. The various races of the domesticated silkworm, *Bombyx mori,* can be roughly divided into those which spin white, yellow or yellow-green silk. Only a few of the many varieties of wild silkworms build white cocoons. The majority of them produces silk the color of which may range from light brown to black brown. The best known examples of brown silks are the "Tussah-Silks" of *Antheraea mylitta* or *Attacus pernyi.* Other wild races spin green silk as for example *Yama mai, Rhodia fugax* or *Theophila mandarina* or golden yellow silk as for example *Cricula trifenestrata.* The natural coloring matters of silk can therefore be roughly divided into yellow, green and brown pigments.

The Yellow Pigments

A. The Chemical Nature of the Yellow Pigments.—Because of their wide distribution among the silks of *Bombyx mori,* the yellow pigments were the first to attract the attention of the chemists. Already in 1808 Roard [62] extracted from yellow silk about 0.02% of a resinous yellow substance which he found to be soluble in alcohol and ether and to melt between 12 and 30° C. This colored "resin" was partly dissolved by alkali and was "bleached to a waxlike substance when treated with concentrated nitric acid." Twenty-eight years later Roard's observations were substantiated by Mulder.[45] In the light of more recent investigations it is obvious that the material isolated by Roard and Mulder was not the pure pigment but rather a mixture pigment and silkwax (Bergmann [6]). The general properties of the pigment, however, as described by these two authors served as the first indication that the yellow coloring matter of silk belongs to the group of lipochrome pigments or carotinoids.

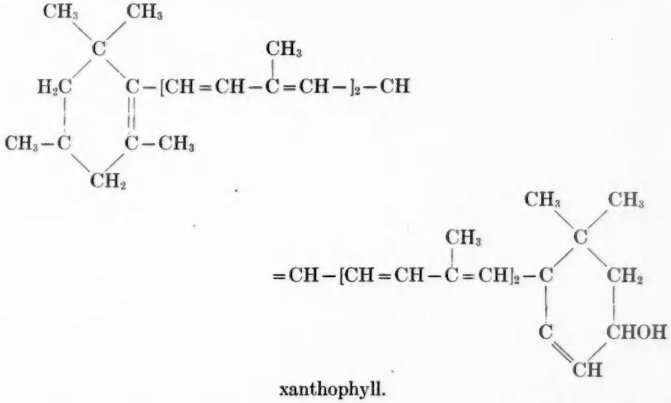
The systemic study of the biological origin and the chemical nature of the yellow pigment began during the latter half of the past century. In order to ascertain the origin of the pigment, Roulin and Sicard [64] examined

* Senior Research Fellow of the Textile Foundation (1938–1939) operating in the Sterling Chemistry Laboratory, Yale University, New Haven, Conn.

microscopically the silk glands of one of the yellow races of *Bombyx mori*. They observed that a few days before the spinning of the silk the contents of the glands were almost colorless. The pigment did not make its appearance in the gland until a few days later and then first in the posterior part of the gland from where it progressed gradually toward the anterior part of the middle section. Roulin and Sicard believed that the pigment was secreted from the walls of the gland and diffused into the liquid silk. About the same time Blanc [6] found that a careful histological examination failed to show any difference between the silk producing organs of the white and yellow races of *Bombyx mori*. He consequently concluded that the pigment was not a product of the gland itself but originated somewhere else. Since silkworms which spin yellow silk invariably possess yellow blood, Blanc at once suspected that there existed some relation between the pigments of blood and silk. On comparing alcoholic extracts of yellow blood, freshly extirpated yellow glands and yellow cocoons, Blanc found that all extracts possessed the same color and showed absorption in the same region of visible light. He therefore concluded that the yellow pigment of the blood and silk were identical substances, and that at some time shortly before the beginning of the spinning process the blood pigment is passed into the liquid silk.

Preliminary studies on the chemical nature of the pigment were carried out by the French physiologist Dubois.[17, 18, 22] He succeeded in isolating from yellow silk a mixture of crystalline substances, which could not be identified because of lack of suitable analytical methods. The results of his experiments, however, convinced Dubois that the pigment was closely related to the "vegetable carotin" which is relatively abundant in mulberry leaves. The discovery of the close relationship between the leaf and silk pigment brought about lengthy discussions on the biochemical origin of the silk pigment. One group of investigators regarded such a relationship as a strong indication for the exogenous origin of the pigment; in other words, they believed that the worms had utilized the leaf pigment for the coloration of the silk. Another group of investigators, however, clung to the belief that the pigment was of endogenous origin, that is to say, it was produced by the worm itself. In the numerous discussions which took place between the adherents of the two groups the results of investigations played an important role which dealt with attempts to obtain colored silk by feeding silkworms natural or synthetic dyes. A detailed description of these experiments will be presented in a later chapter of this report. Suffice to say at this point that because of his failure to obtain colored silk by such feeding methods, Dubois concluded that the worms could not absorb pigments and that the yellow pigment of silk was consequently of endogenous origin. Conte and Levrat [13] on the other hand found that certain dyes could indeed be dyalized through the intestinal membranes of the worm, penetrate into the blood and eventually be fixed in the silk gland. They consequently postulated that the silkworms could absorb plant pigments and that the pigment was of endogenous origin. The reasoning based on the results of such feeding experiments cannot carry any weight in the discussion of the origin of the silk pigment. There exists such a fundamental difference between the chemical and biochemical properties of the dyes used in the experiments and the yellow pigments of leaves that it is not permissible to draw any parallels.

A better argument in favor of the hypothesis of the exogenous origin of the yellow pigment of silk was the observation made by Levrat and Conte [37, 38] that there exists a great similarity between the absorption spectra of leaf and silk pigment. The existence of such a similarity was later denied by Migliardi O'Riordan [47] and Pigorini.[55] It must be kept in mind, however, that deductions which have been built exclusively on comparisons of absorption spectra of mixtures of carotinoids are often misleading and also that the yellow pigment of silk is a relatively unstable substance, which is easily oxidized to products possessing absorption spectra different from the original. Such alleged differences in absorption spectra have been some of the principal reasons why there has been so much reluctance in accepting the theory of the exogenous origin of the yellow silk pigment. In 1923 [1] and as recently as 1930 [2] the prominent Italian investigator Acqua has voiced his doubts about the validity of such an hypothesis. In his book "Il Bombica del Gelso" he has stated "that the theory of direct penetration of plant elements as f.i.xanthophylls (the yellow pigments of leaves) into the blood and silk does not seem acceptable" and "that the pigment of silk must be considered a product of special organs."



xanthophyll.

In 1929 the yellow pigment of silk was first isolated in a pure crystalline form by the Japanese investigator Oku [49] who also identified it as xanthophyll (lutein), a pigment frequently found in organic nature. Besides xanthophyll yellow silk contains small amounts of carotin in various oxidized forms as well as some violaxanthin. The xanthophyll content of normal yellow silk was found by Oku to be 0.022–0.024% and the carotin content 0.0006–0.0008%. The intensity of the color of silk depends on the concentration and combination of the carotinoid pigments. The pink pigments of the "Chinese Rouge" cocoons also belong to the xanthophyll group.[53] Oku [51] also showed that the fading of yellow cocoons as well as the appearance of the "yellow cocoon odor" results from the oxidation of the pigments.

In 1930 Oku [50] proved the identity of leaf and silk pigment by the isolation of xanthophyll (lutein) from mulberry leaves. A few years later

Barbera [4] isolated a crystalline pigment from mulberry leaves which, however, was not positively identified. Oku's discovery of the identity of leaf and silk pigment as well as the fact that pigments of this type are readily absorbed by the animal organism speaks greatly in favor of the exogenous origin of the silk pigment. Although final conclusive evidence is still missing it appears rather certain that the yellow pigments of silk have been derived from those of the mulberry leaf (see Lederer [36]).

It is a well-known fact that the complete removal of yellow pigments from silk by methods other than bleaching is not readily accomplished. Even a prolonged extraction of yellow silk with solvents in which carotinoids are very soluble is rarely sufficient to remove all of the pigment. Dubois [20] found that the pigment could easily be extracted with alcohol if the silk had previously been subjected to a treatment with an 8% aqueous solution of sodium carbonate. Oku [40] obtained his extracts of pigments by heating yellow cocoons with 80% alcohol under 15 pounds of pressure. He concluded that the pigment is physico-chemically combined with the sericin. It remains to be seen whether this assumption is correct. On the basis of the experience of the writer of this report it appears rather certain that the pigment is a product of absorption or addition of a carotinoid to some protein of the silk, and that pigment cannot be readily extracted until such combination has been destroyed.

This phenomenon finds an interesting parallel in the behaviour of the blood pigment of the silkworm toward solvents. Poulton [56] has shown that the yellow pigment of the larval blood of certain Lepidopterae possesses a great stability which he attributed to some combination with the protein of the blood. Ether, carbon disulfide, and chloroform extract the blood pigment very slowly only, while it dissolves instantaneously in alcohol which acts as a protein precipitant. It would be of considerable interest to ascertain whether the transfer of the pigment from the blood stream to the silkgland is a transfer of carotinoids as such or of a carotinoid-protein combination.

Carotinoids can be found in the blood of many silk-producing caterpillars. They have been found for example in the hemolymph (blood) of *Samia cecropia, Telea polyphemus, Saturnia pyri* and *S. pernyi*. Geyer [29] believed to have noticed interesting sex differences in the pigmentation of the blood of *Bombyx mori*. He has claimed that the larval and pupal blood of the male insects is always colorless while that of the females is always deep yellow. This observation, however, has never been confirmed and must have been based on some error. If the blood of the male worms is always colorless as maintained by Geyer, one should not expect male worms to produce yellow silk, which in no way agrees with actual observations.

B. Racial Differences in Yellow Pigmentation.—Racial differences play an important role in the pigmentation of the blood and silk of *Bombyx mori*. As a rule the color of the silk closely corresponds to the color of the blood. But for one exception all races producing white cocoons possess colorless blood. Dubois [22] thought that in contrast to the yellow races of *Bombyx mori* the white races were not able to synthesize the pigment. Since it is, however, almost certain that the pigment is of exogenous origin, this hypothesis is no longer acceptable. Levrat and Conte [37] have attributed the difference in the color of the blood to a difference in the ability of the worm to absorb the leaf pigments. According to these authors white silkworms produce white silk because the yellow pigments of the leaves cannot pene-

trate into the blood stream and the silk gland. During the past ten years this hypothesis has been extensively studied by the Italian investigator Jucci.[33] This author refers to the factor controlling the color of the blood as "the permeability of the intestinal membranes for carotinoids" and the Japanese investigator Uda refers to it merely as factor C.

There exists an exception to the general rule that the color of blood and silk correspond to each other. In 1919 Uda[68] discovered a pure race of silkworms which possess yellow blood but nevertheless produce white silk only. Consequently the "positive permeability" or factor C alone is not sufficient to cause yellow silk. An additional factor is required which has been named by Jucci the "capacity of the silk gland to absorb the pigment" and factor Y by Uda. This factor is of course of no importance in the case of worms with colorless blood, which explains the non-existence of worms with colorless blood and yellow silk. The existence of this factor explains why it is possible to obtain worms producing yellow silk by crossing two white races. This is the case when a white race, which is white because of the absence of blood pigment (normal white), is crossed with a race which is white because of the incapacity of the silk gland to absorb the yellow blood pigment.

In this connection an observation made by Gerould[27] (1921) is of interest. This investigator found that the blue green mutation of the normally grass green caterpillar *Colias Philadice* lacks the normal yellow blood pigments. Hence during the mutation the ability to absorb the yellow leaf pigments has disappeared. There exists an interesting biological relationship between the blood pigment of this caterpillar and the cocoons of the parasite *Apalantes flaviconchae* which emerges from it. The cocoons of parasites which have grown on the normal worm are yellow, while those of the carotinoid free mutant are white. In this case therefore the relationship between the color of the blood and cocoon even extends to the parasites.

Among the silkworms with colorless blood and white cocoons one distinguishes between recessive and dominant white races. If a recessive white race is crossed with a yellow race all the descendants of the first (F_1) generation produce yellow cocoons and in the second (F_2) generation 75% produce yellow and 25% white cocoons. The opposite is true among the descendants of a crossing between a dominant white and a yellow race. In this case all the cocoons of the first (F_1) generation are white and of the second (F_2) generation 75% produce white and 25% yellow silk. It has been concluded (Manunta) that the recessive white race is white because it lacks Uda's factor C or the ability to absorb the pigments, but that the dominant white race is white because of the presence of some inhibiting factor which makes the presence of carotinoids in the blood impossible (factor S). This explanation is related to a hypothesis which has been advanced by Vaney and Pelosse.[71, 72] These investigators found that the blood of white races darkens rapidly when exposed to air while that of yellow races only turns superficially brown. They were confident that the darkening of the white blood was caused by some "oxidizing diastase, tyrosinase" which was secreted by the blood. In the opinion of these authors all silkworms regardless of the final color of their blood absorb the yellow pigments of the leaf. In the case of white worms, however, these pigments are destroyed as soon as they enter the bloodstream.

The value of this hypothesis is somewhat questionable at least as far as the recessive white races are concerned. Whether, as believed by Jucci, it can be applied to the dominant white races is as yet uncertain. According to recent work by Manunta [40] experiments in vitro do not support such a hypothesis. A mixture of the colorless blood of the race "Bagdad White" and deep yellow blood does not decolorize as rapidly as one should expect if the colorless blood contained such a powerful decolorizing enzyme. Manunta demonstrated, however, that there exist considerable differences in the behaviour of dominant and recessive white races toward purpurin. This dye readily stains the blood and silkglands of the recessive white race but is immediately decolorized by the same products of the dominant white race.

As a rule the yellow pigments are unevenly distributed in the various layers of the cocoons. Certain layers may be extremely rich, others poor in pigments and some even colorless. This well-known fact is taken into account during the reeling of yellow cocoons. Different strata of several cocoons are reeled into one thread so that its color represents the average color of the cocoon. In the cocoons of the race "Chinese Gold" the outer layers are of deep yellow color, the middle layers are less pigmented and the inner layer is almost white. In the cocoons of the native Italian yellow race "Fossombrone" the outer layer contains only little pigment, the middle layers are highly colored and the lining again is almost white. According to Jucci [33] the different distribution of pigment in the cocoons of the Chinese and Italian yellow race is caused by a difference in time at which the migration of the pigment into the silk gland takes place. This migration occurs early in the Chinese race, namely between the 5th and 7th day of the fifth age. Consequently the blood of the fully-matured worm is almost colorless. The first silk secreted by the worm during the spinning process is highly pigmented. During the maturation and the spinning process, however, remarkably large quantities of silk are still produced for which there is little if any pigment left. Consequently one notices a constant decline in pigmentation from the outer to the inner layers of the cocoon. In the "Fossombrone" race the gland begins to absorb the pigment only after maturation, so that the first silk leaving the reservoir is almost colorless. With an increase in the rate of migration of the pigment, the color of the silk deepens and decreases again in the last layers. In the case of the "Fossombrone" race only part of the blood pigment is absorbed by the silkgland. The blood of the chrysalis still contains some color. Jucci's interpretation of the uneven distribution of pigments in the yellow cocoons has recently been attacked by Buonocore and Malucelli,[10] who attribute this phenomenon not to a difference in the time of the migration of the pigment, but to a difference in the section of the silk reservoir into which the migration takes place.

By crossing the Chinese Gold and the Fossombrone race Jucci [33] obtained worms which in the first generation produced cocoons which showed a distribution of pigments intermediary to that of the parents. Layers of different colors have also been observed in the flesh cocoons produced by a certain variety of *Bombyx mori*. The usual flesh cocoon consists of a flesh-colored outer and a yellow inner layer. Uda [69] was able to separate this race by inbreeding into a race with pure yellow and a race with pure flesh-colored cocoons.

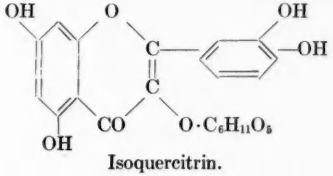
Even in case of the yellow races only a fraction of the carotinoids of the mulberry leaves are absorbed by the worm. The remainder is excreted with the droppings. Attempts to detect quantitative differences in the contents of carotinoids of the droppings of white and yellow silkworms have as yet been unsuccessful (Manunta [43]).

The eggs of *Bombyx mori* are more or less pigmented and there is a certain relationship between the color of the eggs, blood and silk. Races with yellow cocoons also produce yellow eggs, the color of eggs of white races is much paler. Though the yellow pigments of the eggs have not yet been definitely identified, there can be little doubt that they are identical with the blood and silk pigments (see Duce,[24] Jucci,[33, 34] Manca,[39] Manunta,[41] Pigorini,[54] Teodoro [67]).

Certain caterpillars like the tent moth caterpillar *Malacosoma americana* or *Gastropachia neustria* build small pale yellow cocoons. On closer observation one can find that these cocoons consist of a few loosely-woven layers of white silk which have been covered by a yellow, dust-like material. It has been shown by Gerstäcker [28] and others that this dust represents the solid residue of the yellow content of the Malpighian tubes with which the worm has covered the cocoon. The yellow pigment is therefore not the coloring matter of the silk, but that of the urine of the worm. Nothing as yet is known about its chemical nature.

Green Pigments

A. Pigments of the Flavone Type.—Certain Japanese races of *Bombyx mori* spin yellow-green silk. In a series of investigations on this type of silk Jucci and Manunta [35] arrived at the conclusion that the green pigment is closely related to the flavones, a group of pigments which is quite common in the plant world. Jucci and Manunta have named the pigment Bombichlorin, but they did not isolate it in a pure state. About the same time, 1934, Oku [52] isolated the green pigment of the cocoons of the Japanese variety Seihaku of *Bombyx mori*. This pigment, which was named bombycin by the discoverer, decomposes at 300° C. It gives all the typical color reactions of flavones and is a monoglucoside. On hydrolysis with 5% sulfuric acid bombycin is split into glucose and the aglucon bombycetin of m.p. 170–180° C. Bombycetin possesses the composition $C_{20}H_{19}O_7N$ and contains at least three hydroxyl groups. Its absorption spectrum shows maxima at 2700 and 3750 μ and closely resembles that of quercetin. Neither bombycin nor bombycetin have as yet been isolated from mulberry leaves. The leaves contain, however, another flavonol glucoside which was found by Oku [52] to be identical with isoquercetrin.



Isoquercitrin.

It is quite conceivable that the isoquercetrin of mulberry leaves is absorbed by the silkworm and changed biochemically into bombycin. Ac-

cording to Oku [53] and Manunta [42] flavone-like pigments are widely distributed among the various races of *Bombyx mori*, although often in very small quantities only. Color reactions have shown its presence in white and yellow silk, as well as in the blood and eggs of various races. It is also possible that the yellow color of the skin of certain silkworms is caused by the presence of such pigments (Manunta [42]).

The discovery of flavone pigments in the silkworm is of considerable interest because pigments of this type are rarely met with in the animal kingdom. Since flavones turn brown readily in the presence of alkali, their presence in silk assumes practical as well as scientific importance. Manunta [35] has pointed out that it is quite conceivable that the ammonia vapors which are often met with in rearing houses and which are produced by the fermentation of the droppings, may cause a discoloration of the silk.

Uda [69] found that on crossing a dominant white race with a green race worms are obtained which produce green cocoons only. The interpretation of dominant white as "the presence of a factor inhibiting the appearance of color in blood and silk" can therefore only be correct as far as the carotinoid pigments are concerned.

B. Other Green Pigments.—Green silk is also produced by several varieties of wild silkworms, of which the best known is the silk of *Saturnia Yama mai.* The pigment of *Yama mai* was first investigated by Dubois [19] (1890) who stated that it was unevenly distributed on the cocoon in the form of small yellow-green crystals. Later he also described the isolation of two crystalline pigments from *Yama mai,* namely the green chloro-yamamain and the blue cyano-yamamain. Other investigators, however, demonstrated that these substances were not pure compounds. Verson pointed out that the worms of *Yama mai* evacuate certain excrements inside the finished cocoon which on drying leave a crystalline residue consisting of oxalates and uric acid. It is very likely that such crystals, colored by certain impurities, were erroneously regarded as the pigments of *Yama mai* (Conte [12]).

Levrat and Conte [37, 38] observed that the absorption spectra of extracts of the green silks of *Yama mai, Rhodinia fugax* and *Rhodinia newawa* closely resembled that of chlorophyll. They also observed that the silk in the gland as well as the blood of the worms was colored by a pigment of similar type. The authors therefore concluded that the worms in question were able to absorb the chlorophyll of mulberry leaves into the blood and from there into the silk. Dubois and Villard [74] doubted the similarity between the green pigment of silk and chlorophyll, but Gautier [26] and more recently Oku [53] corroborated the observations of Levrat and Conte. Oku concluded on the ground of careful spectroscopic examinations that the green pigment of the silk of *Rhodinia fugax,* which closely resembles that of *Yama mai,* was identical with chlorophyll. The experimental evidence obtained with the green silk of *Theophila mandarina* was insufficient to warrant any definite characterization.

The chlorophyll which is consumed by the silkworm undergoes comparatively small changes during its passage through the digestive system. H. Fischer [25] isolated from the dark green droppings of *Bombyx mori* a crystalline pigment of the formula $C_{34}H_{36}O_6N_4$, the phyllobombycin. He concluded that under the influence of the digestive juices of the worm the chlorophyll, $C_{55}H_{72}O_5N_4Mg$, was deprived of the magnesium, the phytol part and one methyl group, and that it had also undergone slight oxidation. It

is as yet undecided whether the phytol part of the chlorophyll molecule plays a definite role in the biochemistry of the worm, or whether it is excreted unaltered. Marchlewski and Urbanvzyk [40] arrived at somewhat different conclusions. According to their findings the pigment of the droppings, which they refer to as anhydro-β-phyllotaonin, is identical with Conant's phaeopurpurin 18. In general their results, however, agree with those obtained by Fischer.

The Brown Pigments

Brown pigments are widely distributed among the silk of wild silkworms. They differ radically from the yellow and green pigments of silk in that they do not become evident until some time after the completion of the cocoon. Blanc,[6] Levrat and Conte,[38] Dewitz [15, 16] and other investigators have shown that the cocoons of *Antheraea mylitta, Attacus orizaba, Attacus pernyi, Saturnia pyri* and *Saturnia pavonia* are white in the beginning and turn brown 24–48 hours after their construction has been completed. This change of color, which was already known to Reaumur [60] (1736), is caused by an excretory liquid with which the worm thoroughly wets the cocoon. The liquid originates from the anus and represents the last excrements of the worm before pupation takes place. That there actually exists a close relationship between the excretory liquid and the appearance of the brown color has been demonstrated by a simple but convincing experiment. Dewitz [16] sealed the anus of silkworms in the process of spinning by a drop of fast-drying varnish thus preventing further excretion. The result of the experiment was that the silk remained white.

In contrast to the worms of *Bombyx mori* which undergo a thorough purgation before they begin to spin, wild silkworms frequently utilize excrements in the construction of their cocoons. In many instances excrements containing considerable quantities of solid material serve to give a loosely-woven cocoon some additional strength. In other cases they are instrumental in the development of the brown color. Certain *Saturnidae,* for example, after they have ceased to feed and excreted the last droppings, evacuate a thick brown liquid which is brown first but turns colorless later on. Verson [73] believed that it was this liquid which stained the white cocoon brown. This, however, is not the case because the brown liquid is in no way related to the change of color of the cocoon. It is excreted before the worm begins to spin, and the fluid which is actually excreted inside the cocoon is either colorless or light yellow.

Levrat and Conte [38] believed that the excretory fluid contained certain elements of the blood, which turn brown when exposed to air and hence cause the coloration of the cocoon. Their explanation therefore denies any direct relation between the brown pigment and the silk itself. But here again the facts were not reconcilable with such a hypothesis, because the liquid under consideration does not originate in the blood but represents the last contents of the Malpighian tubes. Dewitz [16] moreover has demonstrated that the presence of the excretory fluid is not absolutely necessary for the appearance of the brown color, but that it can be replaced by water or glycerol. The fresh white cocoons of certain wild silkworms turn brown rapidly when placed into water to which air has free access or which contains oxidizing agents. This color change is

impeded by the presence of hydrogen or reducing agents. When treated with boiling water the cocoons remain colorless but the aqueous extracts turn brown. All these observations strongly indicated the presence of a soluble chromogen which turns brown under the influence of enzymatic oxidation. Przibram [57] and Spitzer [65] corroborated Dewitz's statements and concluded that the chromogen was a close relative of the derivatives of ortho-dioxy-benzene. In 1927 Przibram and Schmalfuss [58] isolated pure 3,4-dioxy-phenyl-alanin (dopa) from the cocoons of *Samia cecropia* and proved conclusively that this chromogen, which is widely distributed in the insect world, is responsible for the development of the brown color of the cocoons of certain wild silkworms.

Dyeing of Silk in Vivo

During the past one hundred years many attempts have been made to obtain silk of various colors by supplying the feeding worms with certain dyes. In 1840 Bonafous,[9] inspired by the Chinese method of feeding silkworms rice starch which had been spread on mulberry leaves, fed worms of the fifth age with leaves which had been sprinkled with indigo or madder. He claimed to have obtained blue and pink cocoons by this manner, and his findings were later corroborated by Blanchard [8] and Roulin.[62] Other investigators, however, like Alessandrini [3] (1851) and Joly [31] (1852) demonstrated that such cocoons were superficially colored only and that the silk was merely soiled by particles of the dye, which had adhered to the body of the worm. They found that the silk leaving the gland was quite colorless and that worms which had been thoroughly washed before the beginning of the spinning process produced merely white cocoons. Dubois [18] and Blanc [7] were unsuccessful in obtaining colored silk by feeding silkworms about thirty different anilin dyes. Although some of the dyes, as for example fuchsin, were readily absorbed by the worm and deposited in various organs, none of them penetrated into the silk gland.

Conte and Levrat,[12] however, found that neutral red is freely, and methylene blue somewhat reluctantly, absorbed by the worms and deposited in the silk gland where the liquid silk becomes stained. Similar observations were made by Quajat [59] who found that Sudan III is also readily absorbed and does not only pass into the silk but also into the egg. In more recent years the feeding experiments have been taken up again by several groups of investigators. Vaney and Pelosse [70] (1922) have demonstrated beyond doubt that worms of the fifth age can absorb and pass on to the silk gland certain dyes, as, for example, neutral red and orcein. Similar results were also obtained by Jucci and Ponseveroni.[32] Couvreur and Clement [14] tried to obtain colored silk by injecting solutions of dyes into worms ready to spin. They found, however, that only body tissues could be stained by this method. Probably the best qualitative and quantitative method for the administration of dyes to silkworms either by mouth or by injection has been described by Campbell.[11] This investigator suggests the use of certain coal tar dyes for the identification of worms used in biological assays.

In summing up the investigations concerning the staining of silk in vivo, it can be said that, while they are of a certain scientific interest, they possess little if any practical value for the sericulturist.

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A Study of Warp Size Distribution on Continuous Filament Bright Viscose Rayon

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Summary

Evidence obtained by means of a study of cross-sections of sized rayon yarn is presented to show the marked difference in distribution on the yarn between the gelatin-glue class of size and the converted starch class. The effect of relative viscosity of the size mix is illustrated. The different behavior of yarns from different manufactures is noted, and the effect of can-drying on the yarn cross-section is discussed.

Introduction

THE examination under the microscope of cross-sections of sized yarn affords a technique for determining the distribution of size on the yarn and leads to a study of the factors which affect this distribution. The details of such a method and a means of obtaining permanent records of observations in the form of photomicrographs were presented in a previous article of this series. (TEX. RSCH., Vol. IX, No. 8, Pp. 285–291, June 1939.) The present article deals with applications of the method.

Limitations of the Photomicrographic Method

Before presenting applications, however, it would be well to stress some of the limitations to the usefulness of this method. Three of these are: (1) the difficulty of obtaining a representative sample; (2) the difficulty of estimating the amount of size present; (3) the time and skill required on the part of the operator for the production of sections and photomicrographs of them.

(1) When one considers the amount of yarn actually sectioned and viewed under the microscope and compares it with the amount of yarn in even a small warp, it is readily seen that the probability of obtaining an absolutely representative sample is very small indeed. The possibility may be increased slightly by taking the yarn from widely separated places in the warp rather than from the same locality. One must also be very careful

* Dr. Yelland was director at Massachusetts Institute of Technology of the filament viscose rayon phase of the warp sizing research from Jan. 1, 1936, to Sept. 30, 1938.

to keep an open mind and not attempt to form rigid conclusions from a small number of sections.

(2) The sections and photomicrographs should be used only as a means of studying the distribution of the size and not as a means of estimation of its amount. Slight variations in thickness from section to section may create false impressions as to the relative amounts of size present in different samples. The depth of blackening on a photographic print depends on too many factors in the process of photographing and developing to be relied on as a quantitative measure of the amount of size present. All estimates should therefore be made on the basis of desizing experiments.

(3) A study of the details of the process as outlined in the previous article will show that it is a difficult and time-consuming procedure and is best attempted only by those already familiar with photomicrographic work.

Comparison of Starch and Gelatin Distribution

Perhaps the first fact revealed by this technique was the very marked difference in the behavior of the two general classes of size materials, namely glues and gelatines, as contrasted with modified starches of various types. It is well understood, of course, that a wide range of products is covered by the terms "glues and gelatins," and that there are many varieties of modified or converted starches, depending on the kind of starch used and the type and degree of modification. Consequently, not all the members of each class behave in the same manner. However, it may be stated as a general rule that, when they are applied to bright viscose filament rayon yarn from size baths of the same viscosity, gelatins and glues tend more to coat the yarn as a whole while modified starches tend to penetrate between the filaments and to lie mainly in the interior of the yarn in rather irregular masses between groups of filaments, and in the small grooves or crenulations of the filaments. There is not much tendency for the modified starches to form complete films around the individual filaments. In more viscous size mixes the yarn as a whole may be partially surrounded by a rather thick film of starch. Gelatin on the other hand appears to be present in larger amounts on the exterior of the yarn in the form of films which may or may not be continuous around the entire yarn. Only in very fluid mixes does it penetrate much into the interior of the yarn.

This difference is illustrated strikingly in samples sized with a mixture of starch and gelatin (see Fig. 1, A and B). These samples were taken from a warp sized with a mixture containing in 100 finished gallons 8.75 lbs. chlorinated tapioca starch, 8.75 lbs. of edible gelatin and 3.75 lbs. glycerin. It was not feasible to treat the same sections to show the presence of both starch and gelatin at once. So one set of samples was carried through the usual tannic acid treatment for gelatin. A typical section is shown in A of Fig. 1. A second set of samples was treated according to the method for starch, and yielded sections similar to Fig. 1, B. A comparison of these photomicrographs shows the marked contrast in behavior of the starch and gelatin size materials.

This difference in starch and gelatin distribution may also be observed by comparing the two series of photomicrographs in Figs. 2 and 3 which illustrate the effect of size viscosity on distribution.

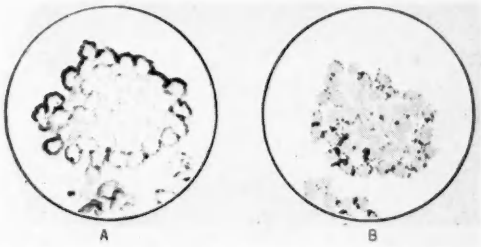

FIG. 1. Photomicrographs of cross-sections of 150 denier 40 filament bright viscose rayon yarn taken from a warp slashed in the mill with a 50–50 size mix of modified tapioca starch and edible gelatin, illustrating the difference in size distribution of modified starch and gelatin. Photomicrographs taken by reflected light. Embedding medium is gum arabic.

A. Yarn treated with tannic acid before sectioning to show the distribution of the *gelatin* only. B. Yarn treated with iodine after sectioning to show the distribution of the *modified starch* only.

Effect of Size Viscosity

When the viscosity of the size mix is increased by increasing the concentration of the size material and all other factors (size, temperature, squeeze roll pressure, speed of slashing, etc.) are kept constant, two results are evident: (1) the amount of size taken up by the yarn is increased; (2) the size tends to coat the outside of the yarn more and to penetrate less between the filaments in the interior of the yarn.

The converse of these statements is also true, of course; that is to say, when the viscosity of the size mix is decreased by decreasing the concentration of size material in the size mix, less size is taken up by the warp and there is a greater tendency for the size to penetrate between the filaments, and less tendency for the size to coat the yarn as a whole. The first result is known probably to everyone who has supervised the process of slashing. It is illustrated by Table I.

The per cent size on the yarn was determined by desizing. The value given in the table is the average of several determinations.

TABLE I

Type of Yarn	Pounds of Gelatin per 100 finished gallons of size	Pounds of Sulfonated Olive Oil per 100 finished gallons of size	Viscosity of Size Mix Relative to viscosity of water at 60° C.	Per cent size on Yarn (Average Value)
A 150/42 Bright	16.25	7.5	1.8	3.5
B 150/42 "	22.25	10.	2.4	5.9
C 150/40 "	20.	10.	2.2	4.0
D 150/40 "	22.45	10.	2.4	5.1
E 150/40 "	27.5	10.	2.8	5.9

The second result is illustrated by the photomicrographs in Fig. 2, A, B, C, D and E. In these can be seen the cross-section of one entire yarn with the edges of the cross-sections of neighboring yarns. The yarn section visible in each photomicrograph is only one of 20 or 30 sections embedded in a gum arabic * section. Hence the gum arabic forms the gray filament boundaries can just barely be detected where there is no gelatin. Where the gelatin is present it has been rendered opaque by precipitation

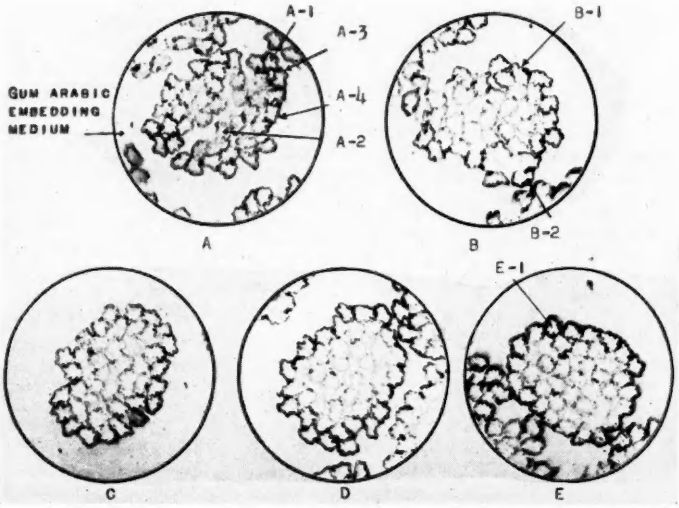

FIG. 2. Photomicrographs of cross-sections of continuous filament bright viscose rayon yarn, sized in the mill with gelatin size mixes at various concentrations, illustrating the effect of increasing size viscosity. Photomicrographs taken by reflected light. Embedding medium is gum arabic. A and B, 150 denier 42 filament, with relative size viscosity of 1.8 and 2.4 respectively; C, D and E, 150 denier 40 filament, with relative size viscosities of 2.2, 2.4 and 2.8 respectively.

with tannic acid and can be seen as dark irregular masses. The gum background which is visible between each section and its neighbor. The arabic forms a matrix in which the filaments are embedded. This ma-

* The sections from which the photomicrographs in Figures 1, 2, and 3 were made, were cut from yarn embedded in gum arabic rather than in methyl methacrylate resin. The use of gum arabic embodied an older and more cumbersome technique than the use of the resin. When these photomicrographs were made the details for the resin method had not been worked out completely. The gum arabic method was given up as soon as it was found that the same results could be achieved more quickly and conveniently by the use of methyl methacrylate resin. Details of the older technique are therefore not included in these articles. They can be found in several well known texts, e.g., E. R. Schwarz, "Textiles and the Microscope," McGraw-Hill Book Company, N. Y., 1934; J. M. Preston, "Modern Textile Microscopy," Emmott and Co., Ltd., London, 1933. Observations in this article referring to gum arabic can be applied equally well to methyl methacrylate resin.

trix extends to a considerable distance beyond the periphery of the individual yarns. Thus the sections which are later cut and viewed show the filaments undistorted and still embedded in the gum arabic. This condition prevents the possibility of securing a contrastingly visible edge of gum arabic around the filaments because of a difference in refractive index between the mounting medium and the gum arabic itself. In the photomicrographs presented in this report the outer edge of the gum arabic is not seen as it lies some distance outside the field of the photomicrograph.

In photomicrographs A and B of Fig. 2 some filaments are coated with a thin film of size (see A–1). Other filaments are almost free of size (see A–2), while the majority have small amounts of size lying in the crenulations of the filaments and in the interstices between the filaments. There is no continuous film of size surrounding the yarn. In A there is a film which is continuous for only the short distance between the points marked A–3 and A–4. In B the film is continuous for a longer distance between the points marked B–1 and B–2.

In photomicrographs D and E of Fig. 2 there is an entirely different condition. The size has formed a continuous film around the entire yarn. This film is quite heavy in E. Only a very few filaments (see E–1) have an individual coating of size. A small amount of the size in E is distributed within the interior of the yarn. C of Fig. 2 represents a condition intermediate between the extremes shown in A and E.

A similar effect of viscosity on penetration and coating may be seen in photomicrographs of starch-sized yarn shown in Fig. 3. These are samples which were taken from different mills and hence were sized on different slashers under widely different conditions. Therefore they do not form a related series as did the gelatin photomicrographs.

These photomicrographs were made according to the directions given in the previous article already mentioned. The iodine treatment colors the starch an intense blue-black, and the viscose a shade of yellow or orange. The gum arabic is stained very little. When these sections are photographed the starch-iodine compound contrasts more vividly with the viscose than did the "gelatin-tannate" in the preceding sections. It is therefore possible to use a shorter time of exposure, and the background, consisting of gum arabic as before, is often a lighter gray than in the gelatin pictures.

Table II gives the data relating to the size mixes from which these were sized. It must be borne in mind that since these samples were

TABLE II

Type of Viscose Yarn (Bright)	Pounds of Starch per 100 finished gallons of size	Pounds of Softener per 100 finished gallons of size	Viscosity of Size Mix Relative to Viscosity of water at 60° C.	Per Cent Size on Yarn
F 150/48	35.0 converted tapioca	6.7 sulfonated oil	1.3	6.1
G 150/48	37.3 chlorinated "	6.7 glycerin	1.7	4.7
H 150/40	37.5 " "	7.5 "	1.7	5.4
I 150/48	46.7 " "	8.6 "	2.0	8.0
J 150/40	87.5 converted "	17.5 sulfonated oil	3.2	9.8

sized at different mills under different conditions, variables other than viscosity are present. Hence the per cent size on the yarn depends not only on the size viscosity, but also on the speed of the slasher, squeeze roll pressure, etc. However, the size distribution can be illustrated by these samples.

In the photomicrographs in Fig. 3 F and J represent the most extreme cases, F having been sized from the least viscous mix (relative viscosity 1.3) and J from the most viscous mix (relative viscosity 3.2). G,

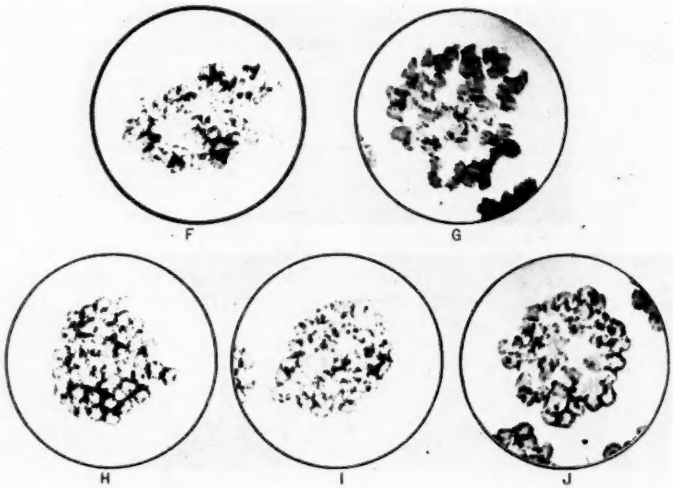

FIG. 3. Photomicrographs of cross-sections of continuous filament bright viscose rayon yarn sized in mills with starch mixes at various concentrations, illustrating the effect of increasing the relative viscosity of the size mix. Photomicrographs taken by reflected light. Embedding medium is gum arabic.

F—yarn, 150–48; rel. size viscosity, 1.3. G—yarn, 150–48; rel. size viscosity, 1.7. H—yarn, 150–40; rel. size viscosity, 1.7. I—yarn, 150–48; rel. size viscosity, 1.7. J—yarn, 150–40; rel. size viscosity, 3.2.

H and I show three intermediate cases. J is the only starch sample which shows any indication of having a continuous film around the outside of the yarn, and even here the film extends only part way. In the samples sized with less viscous mixes, there is no continuous film around the outside of the yarn or even of the filaments; the size lies in discrete masses in the crenulations of the filaments and in the interstices between the filaments. Thus the starch sized samples exhibit, to some extent, the same effect of increasing viscosity of size mix; namely an increasing tendency toward film formation around the outside of the yarn and a de-

creasing tendency toward penetration between the filaments into the interior of the yarn.

Alternative Techniques

A question was raised in regard to the technique in preparing photomicrographs of samples such as Fig. 2, E. It was stated in the preceeding article already referred to that the gelatin sized yarn was treated with tannic acid first and sectioned afterward. It was possible that the "gelatin-tannate" was precipitated as a film around the yarn and was not allowing the tannic acid solution to penetrate into the yarn to react with any gelatin that might be present there.

To invesigate this a sample was prepared, treated with tannic acid, sectioned, and fixed on the slide in the usual way. Then representative sections were photographed. The cover glass was carefully removed, and the immersion oil was washed off the slide with xylene. This left the yarn sections still on the slide. These sections were then treated in situ by immersing the slide in tannic acid solution, alcohol and xylene in the same way in which the original yarn had been treated. Immersion oil was again added to the specimen, the cover glass mounted in place, and sections photographed.

It was hoped that it would be possible to obtain pictures of the same sections before and after the second treatment. Comparison of these would show whether or not the second treatment had precipitated any gelatin which had not been reached by the first treatment. However, the sections which were photographed once invariably washed off the slide during the second treatment and hence could not be photographed again. So we were forced to be content with several pictures taken from the same slide (but not the identical section) after a second tannic acid treatment.

The second treatment did not reveal any more size to be present than was shown by the first treatment. Observers were not able to distinguish between photomicrographs taken from the same slides before and after the second treatment. This indicated that the tannic acid treatment of the specimen before sectioning was adequate and that no gelatin remained in the interior of the yarn which had not had an opportunity to react with the tannic acid. Thus the treatment of specimens in yarn form seemed to be adequate.

In preparing starch sized specimens for sectioning by the older method of embedding in gum arabic, there was always to be considered the possibility that a very thin film of starch might have been dissolved off the outside of the yarn in the gum arabic solution during the embedding process Hence, as soon as the technique using methyl methacrylate resin was available some of the earlier work was repeated and a careful comparison was made of sections cut from the same samples by two different methods. There was no appreciable difference in the appearance of sections thus prepared, and the resin embedded sections gave no indication of any thin continuous starch film around the outside of the yarn. That the resin had no appreciable solvent action on the starch was proved by embedding starch granules in the same way and observing their appearance after polymerization of the resin.

Additional evidence against the loss of starch in the gum arabic method was noted, in that the gum arabic solution remaining in the immersion

bottles after repeated use did not appear to contain any dissolved starch. Also the gum arabic immediately surrounding the starch sections gave no starch test with iodine when the sections were stained. All these confirmatory observations make it appear reasonable that there is no loss of even a thin film of starch size during the embedding and sectioning process.

Comparison of Behavior of Yarn of Same Denier but from Different Companies

Reference to Table I will show that B and D were sized with similar size mixes under as nearly the same conditions as possible. B is a 150 denier 42 filament bright yarn from one company; D is a 150 denier 40 filament bright yarn from a second company. Under nearly identical mill conditions B has picked up 0.8% more size than D. A comparison of the corresponding photomicrographs will show that B is an open, rather irregular shaped section, while D has a more compact oval shape. In B the size has penetrated more into the interior of the yarn than in D although the viscosity of the size mixes was the same.

A study of this difference in behavior of yarn of the same denier but from different sources will doubtless help to remove some of the uncertainty in sizing. Such a study must also take into account differences in the kind and percentage of finish on the yarn, and, in the case of dull and semi-dull yarns, the material used to deluster the yarn, whether it is included in the viscose itself or applied to the yarn surface.

Limit of Size Penetration

Examination of a large number of cross-sections of sized yarn shows, as one might expect, that there is *no* penetration of size material *into* the filaments. The size may or may not penetrate into the yarn between the filaments, but does not penetrate into the filaments themselves.

Effect of Can-drying on Shape of Yarn Cross-Section

There are two methods of drying a sized warp: either by passing it over revolving heated cans, or through a chamber of warm, dry air. The first method is used at present to a much greater extent in this country for synthetic yarn than the second. Can drying has been accused of having many bad effects on the warp. One of these arguments frequently encountered is that can drying deforms the yarn by flattening it. A study of the cross-sections of can-dried yarns shows that perhaps this has been overemphasized.

The warp yarns, from which all the photomicrographs in this report, except Fig. 4, were made, were all taken from warps after can-drying and before weaving. Of these F in Fig. 3 shows the highest degree of deformation from a circular cross-section. The other sections range in shape from rather wide ovals to almost perfect circles.

Let us compare the shape of these sections with sections taken from the cloth after it has been desized and finished. The photomicrograph in Fig. 4 was made from a section taken from a finished warp across the warp ends parallel to the filling. This section was cut 50 microns thick in order to obtain a whole filling yarn. The picture was taken at a magnification of

50 diameters, instead of the usual 200 diameters, in order to show several warp ends at once. The section was *not* treated to show the presence of size material, and was embedded in resin rather than gum arabic. Here the dark lines appearing at the filament boundaries are the actual filament boundaries and *not* size material. In comparing Fig. 4 with the preceding photomicrographs one should compare only the shape of the yarn cross-sections.

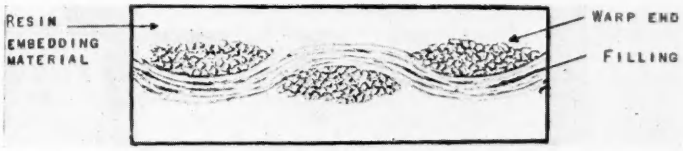

FIG. 4. Filling-wise cross-section of fabric showing flattening of warp yarns in fabric. The dark lines at the filament boundaries are the actual filament boundaries and *not* size material.

As yet there have not been available for this study samples which have been taken from warps which have been air-dried to compare with the can-dried samples. Therefore, although it had not been definitely proven, it is indicated by the results obtained thus far that the deforming effect of can drying has been over-estimated. The yarn is flattened more during weaving and finishing than in can drying.

Acknowledgments

All of the photomicrographs illustrating this article were made by Mr. H. H. Broadfoot, research associate on the staff of the Warp Sizing Project at M. I. T. The author wishes to express to Mr. Broadfoot his appreciation of the skill and meticulous care required in the preparation of the photomicrographs. He also wishes to thank Prof. E. R. Schwarz of M. I. T. for his generous loans of equipment for photomicrographic work, and his helpful advice on methods and techniques.

(*Continued from page 396.*)

Studies on the Developing Cotton Fibre. I. The Relation of the Development of the Crude Cotton Fibre to the Other Principal Boll Constituents, by Jack Compton and Forrest E. Haver, Jr., Chemical Foundation research associates at Boyce Thompson Institute for Plant Research.

Thursday P. M.—The Effect of Temperature and Concentration of Sodium Hydroxide on the Mercerization of Cotton as shown by X-ray Diffraction Analysis, by Wayne A. Sisson and William R. Saner, Chemical Foundation research associates at Boyce Thompson Institute for Plant Research; The Rôle of Enzymes in the Manufacture of Cotton Textiles, by Walter M. Scott of Gustavus J. Essellen, Inc.; Physical Tests for Quality in Textiles, by Herbert F. Schiefer, research associate at National Bureau of Standards.

Drying Cotton Below Normal Regain is Wasteful

"Cotton may be dried fairly quickly at temperatures over 200° F., and the air-water vapor mixtures would have to be very heavily charged with water at these elevated temperatures for the relative humidities to approach values normal at ordinary temperatures. Normally, the R.H. at the elevated temperatures will be very small and there is no need to dry the cotton until it is in equilibrium with the drying air as this would bring the moisture content down to a value below the regain at normal temperatures, and water would have to be added to speed up the normal regain. The stock may be removed from the drier after a much shorter time than that required to bring about equilibrium; it could be brought off the drier at an elevated temperature when containing slightly more moisture than the normal regain. At the temperature of removal, the stock may feel damp, but as it cools to atmospheric temperature, it will dry to normal regain and feel quite well-conditioned. Drying to below the normal regain involves a waste of heat as well as requiring the apparently superfluous process of re-wetting the stock." From "Some Principles of Drying," by W. Cowen in *J. Soc. Dyers Col.*, June 1939.

* * *

The Felting (?) Properties of Casein

TWO of the papers on casein fibre presented at the annual conference at Bath, Eng., in June of The Textile Institute referred to the so-called "felting properties" of that fibre and advanced theories as to the cause of the effect produced. The paper by Diamond and Wormell had the following statement: "The felting properties of the casein fibre have called for some thought, since although it has neither scales nor disulphide links, yet fabrics containing percentages of casein fibre show greater felting properties than some wool fabrics. The felting of casein fibre has been experienced both in acid and alkali medium. In attempting to explain the felting properties of casein fibres, the adhesiveness which is shown by these fibres after processing in hot liquors should be taken into account. This adhesiveness is not deep seated and is readily broken down by working the dry fibres. It may be the result of some superficial hydrolysis of formaldehyde from the surface layers, leading to a gel-like surface, in the same way that felting of wool is explained by some workers as a change in the cuticle layer."

The paper by G. Heim has a similar explanation of the so-called felting of casein, and indicates that it is a temporary phenomenon. It is as follows: "It is generally agreed that felting depends on the surface scales of the fibre and it is therefore to be expected that the casein fibre will not show any felting. It has been stated, however, that it would be possible to felt casein fibre, but possibly this so-called felting may be something quite different. Casein fibre which is not sufficiently hardened will become sticky in hot liquids. Consequently the fibres will stick together at the points where they cross each other and are subjected to pressure. It is to be feared therefore that as the hydrophilic nature of the fibre is decreased, which is necessary to improve the mechanical properties in the wet state, this apparent felting ability will disappear."

Science and Empiricism

The new moisture content controller and indicator for slashers and tenters developed by the Barber-Colman Co., Rockford, Ill., are really the by-products of research conducted by that company for several years at Massachusetts Institute of Technology. The parent research, which we believe is still in progress, has for its objective the use of electricity in determining the grade and spinning properties of textile fibres. Indication and control of moisture content of the fibres are essential factors, and the by-products noted are the first practical results of the research. The study, by the way, involved much fundamental work.

* * *

Fire-resistance of treated cotton ducks and felts has been tested by a new method developed by the National Bureau of Standards in which a 7- by 1-in. sample of a vertical edge is exposed to the flame. The testing method and recommended requirements are incorporated in a report to the Committee on Fire Proofing and Preservative Treatments of the National Fire Protection Association. Federal Specification CCC–D–746, entitled "Duck, Cotton: Fire, Water, and Weather Resistant" contains the flame-proofing requirements.

* * *

The electron microscope developed in the laboratory of electron optics of Siemens and Halske, is said to have a magnifying power of over 30,000, *i.e.*, about 15 times that obtainable with ordinary microscopes. Such a magnification renders visible colloidal forms of dispersed matter and other large aggregated molecules. The new microscope is operated with an electron beam, *i.e.*, electron rays of wavelengths down to 10^{-9} mm. according to the voltage used, and a system of magnets, termed "magnetic lenses." The principle involved is analogous to that underlying the use of the cathode-ray tubes in television receivers.

* * *

Commenting on nylon, Ellis Clayton, co-editor of the *Journal of the Society of Dyers and Colourists* (Jan. 1939, P. 34) says: "It is to be expected that, in view of the remarkable physical and chemical properties of the raw material, many other extruded forms of it will be made ultimately for use in various branches of industry, while its possible use as a base for the manufacture of coating and other finishing materials opens up other fields of application. Finally, it is obvious that the new technique now disclosed will be a powerful stimulus to research attracted towards the synthesis of new types of aliphatic compounds which can be condensed to high molecular complexes without undergoing cyclization."

* * *

"**Mazein**" is the trade name selected by Corn Products Refining Co. for its patented zein products, one of which patents, No. 2,156,929, covers a

method for producing protein filaments from zein solutions. The latter are extruded into a coagulating fluid while the zein is contacted with a reactive aldehyde, and the resulting filaments then subjected to elevated temperatures. When a suitable plasticizer is incorporated it is claimed that the resultant filaments will generally be found to have wet strength, elastic recovery and abrasion resistance superior to the artificial cellulose fibres previously employed. Zein is a constituent of Indian corn and described as "a prolamin allied to glutin obficient in the amino acids tryptophan and lysine."

* * *

Kapok is one of the lightest and "silkiest" of vegetable fibres but lacks all of the properties that render vegetable, animal and synthetic fibres spinnable. However, according to B. J. Perkins, foreign correspondent of *Daily News Record,* Les Fils Textilor, a French corporation, controls patents covering a process for the preparation and spinning of kapok, and he reports that the kapok yarn has been woven by a number of French mills with and without other types of yarns. Kapok's principal use has been as a filling material for bedding, and also as a filler for life-preservers—the latter because of its lightness and its resistance to wetting. In its growth and appearance it is very similar to the fibre in the common milkweed pod, and if it is spinnable commercially the latter fibre should be.

* * *

The chlorination process of the British Research Association for the Wool Industries for rendering wool shrinkage-resistant has been trade marked "Woolindras," and is reported in use on a commercial scale. The annual report of the Association states that "an entirely new method of producing non-shrinkable wool is under investigation. In this process the use is made of certain 'enzymes' which act on protein. These are completed substances present in living cells. One type suitable for the process is papain, a substance present in the milky juice of the tropical fruit called papaya. The activity of the substances can be controlled chemically and they appear to have a specific action on certain regions of the wool fibre." The report states, however, that it is too early to speak of the special advantages of this new type of treatment.

* * *

Dipotassium phosphate solution, developed by the Monsanto Chemical Company for wool lubrication, is claimed to have shown its superiority to olive and mineral oils in spinning tests, and is of particular interest because it is water soluble, is very stable chemically, and has no tendency to oxidize nor to catalyze oxidation reaction. Because of its water solubility there is a marked saving of soap in the scouring process, and when used on scoured wool it can remain on the wool until dyed. It acts as a leveling agent in the dye bath, but as its pH in a 60% solution is 9.9–10.3 a little extra acid must be added to the dye bath. It does not cling as readily to wool as do the oils, best results being obtained when it is applied as a 30% solution—diluted with an equal weight of water. Being a new chemical for a new use, the mill tests to date have indicated the need of certain minor adjustments in the product, and modification of handling procedure, but in general the claims for the product have been substantiated.

Abstracts

Committee on Abstracts and Bibliography:
E. R. Schwarz, Chm., Dr. E. J. Saxl, C. H. Clark.

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Address, C. H. Clark, Sec., 65 Franklin St., Boston, Mass.

I. Fibres (Synthetic and Natural) and Fibre Analysis

Albuminoid Fibres: Production. A. Deglun. *Rusta*, 1938, V. 13, P. 421–25, 467–71; abs. in *J. T. I.*, March 1939, P. A147.

The Ferretti process for the production of casein fibres and a Dutch modification involving treatment with synthetic tanning agents are briefly discussed, earlier attempts to produce fibres from albuminoid materials are reviewed, and a short account is given of two recent Italian processes for the production of fibres from gelatin and other animal and vegetable products, and keratin, and mixtures of these with viscose. (C)

Cellulose Acetate Artificial Silk Industry: Birth, Development and Present Position of—in England. H. Dreyfus. *J. Soc. Dyers Col.*, March 1939, P. 116–24. (C)

Cellulose Acetate Staple Fibre: Resistance to Carbonizing. *Mell. Textilber.*, 1938, V. 19, P. 569–76; abs. in *J. T. I.*, Nov. 1938, P. A668. (C)

Cellulose and Protein Fibres: Structure and Properties. O. Mecheels. *Mell. Textilber.*, 1938, V. 19, P. 579–81; abs. in *J. T. I.*, Nov. 1938, P. A693.

The relations between the structure and strength of rayon filaments are discussed and the advantages of the author's wear test for the evaluation

of such filaments are pointed out. When regenerated cellulose rayons are compared by this method the order of decreasing resistance to wear corresponds to the order of increasing degradation of the raw material used for the production of the filaments. The relation between the degree of degradation of the raw material and the ordinary breaking strength is not so clearly marked and the wear test may reveal differences in quality in filaments of the same breaking strength. Differences in the molecular forms of proteins from different sources are discussed and it is pointed out that the efficiency in use of protein fibres decreases as the protein molecules become shorter and more spherical. Fish protein and casein are compared, fibres spun from them are compared with wool, and X-ray diagrams are given. Space diagrams showing the relations between load, extension, and time show that artificial protein fibres have abnormally high extension and low powers of recovery compared with the natural fibres. (C)

CELLULOSE AND LIGNIN: BACTERIAL FERMENTATION. E. Berl and W. Koerber. *J. Am. Chem. Soc.*, 1938, V. 60, P. 1596–8; abs. in *J. T. I.*, Nov. 1938, P. A702.

It is reported that anaerobic and aerobic bacteria that readily decompose cellulose do not attack cellulose humic acids, lignin, and lignin humic acids. (C)

CELLULOSE FIBRES: ACTION OF LIGHT ON. H. J. Henk. *Mell. Textilber.*, 1938, V. 19, P. 730; abs. in *J. T. I.*, Dec. 1938, P. A761.

Exposure of cellulose to ultra-violet light for a short period results in a polymerization which is shown by an increase in strength and a decrease in copper number. On exposure for longer periods an alkali-insoluble oxycellulose is formed by the splitting off of carbon dioxide and at the same time there is a separation of water as a result of oxidation of the hydrogen of the cellulose. The splitting off of carbon dioxide then decreases and an alkali-soluble oxycellulose containing reducing groups is formed. Lustrous fibres are less readily attacked than matt fibres, Egyptian cotton less readily than Indian cotton, and mercerized cotton less readily than unmercerized cotton. The oxidation of cellulose exposed to ultra-violet light may be accelerated by the presence of certain catalysts, such as the oxides and peroxides of heavy metals. Cellulose fibres dyed with certain dyes are oxidized more rapidly than the undyed fibres although the dyes themselves are fast to light. This effect is attributed to the formation of active unstable molecular compounds between the dyes and the first oxidation products of cellulose. A similar sensitizing action is shown by compounds of uranium. Materials dyed with other classes of dyes may be damaged by attack by the products of photolysis. The greatest reductions of strength produced in this way have been observed with basic dyes, the effect often being increased by the simultaneous decomposition of the mordant. Both tannin and Katanol O are decomposed on exposure to ultra-violet light and cause a loss in strength of the mordanted cellulose materials. The effect of sulphur dyes varies. The most injurious effect is shown by sulphur black which liberates sulphuric acid on oxidation in light. Certain sulphur dyes have a protective action on cellulose fibres. Vat dyes of the anthraquinone series and substantive dyes, especially diazo dyes, also protect cellulose fibres from oxidation on exposure to light. (C)

CELLULOSE FIBRES: MECHANISM OF TENDERING. R. Haller. *Bull. Fed. Inter. Assoc. Chim. Text. et Couleur*, 1938, V. 3, P. 137–56; abs. in *J. T. I.*, Dec. 1938, P. A762.

The tendering action of acids and oxidizing agents on cellulosic fibres is discussed, with particular reference to evidence provided by the microscope, and an attempt is made to find a mechanism in accordance with the facts. The Nägeli and Wiesner conception of micelles of cellulose embedded among micelles of a more reactive material (*e.g.*, pectins and other hemicelluloses) is combined with the Frey-Wyssling conception of long-chain molecules. (C)

II. YARNS AND FABRICS

COTTONIZED BAST FIBRES: PRODUCTION AND USE. *Klepzig's Textil-Z.*, 1938, V. 41, No. 44, P. 1–3; No. 45, P. 2–4; No. 46, P. 2; abs. in *J. T. I.*, March 1939, P. A146.

A general discussion of developments in the production and use of cottonized bast fibres, especially cottonized hemp, in Germany, the appearance, spinning capacity and other properties of the fibres, materials made from mixtures of cottonized bast fibres with cotton and staple fibre now being shown by various German firms, and measures taken to encourage the use of cottonized bast fibres in Italy, Poland and Germany. Production of cottonized hemp in Germany amounted to about 2¼ million kg. in 1938 and it is estimated that production in 1939 will be about 5 million kg. (C)

DESIGN OF TAPPETS AND CALCULATION OF AVAILABLE SHED IN PLAIN WEAVING. R. McK. Kilgour and H. Boffey. *J. T. I.*, Nov. 1938, P. T253–T257.

Describes a method of estimating the shed available for the passage of the shuttle for any loom, carried out in a way which allows of the available shed being quickly estimated for any particular timing of shed with respect to sley movement, once the essential measurements from the loom have been put into graph form. He also emphasizes one or two necessary details in the design of a pair of ordinary, plain shedding undertappets. (C)

DRYING MACHINERY: EFFICIENCY. F. Kershaw. *Ind. Eng. Chem.*, 1938, V. 30, P. 1115–18; abs. in *J. T. I.*, Jan. 1939, P. A31.

A general review of the use of drying machinery, with illustrations of various types. Among references to the diversity of drying problems it is said that rayon skeins with oil, wax or gum dressings take 2–4 times longer to dry than skeins without such dressings. (C)

FELTING PROPERTY OF WOOL: INFLUENCE OF ADMIXED STAPLE RAYON ON. G. Nitschke. *Kunstseide u. Zellwolle*, 1938, V. 20, P. 251–53 (through *C. A.*, 1938, V. 32, Col. 6871); abs. in *J. T. I.*, Dec. 1938, P. A763.

Wool was felted with 30 and 50% of viscose, cuprammonium, acetate and lanital staple fibres under standard conditions and their average tear factor in kg./g. of felt was determined. Compared with pure wool at 20.2 kg. the viscose, cuprammonium and lanital in 30% admixture had values of

11.0–23.6 kg. and for the 50% mixture 7.5–17.8 kg. Fibres with smooth surfaces gave the lower values. The two acetate fibres tested gave low tear factors although their other properties made them especially suitable for mixing with wool. (C)

FINISHING TREATMENTS: TECHNICAL EVALUATION OF. E. R. Schwarz. *Am. Dye. Rptr.*, Mar. 20, 1939, P. 138–46; *Text. Rsch.*, April 1939, P. 216–30. (C)

HIGH-DRAFT SYSTEMS: CHARACTERISTICS. G. Krauter. *Mell. Textilber.*, 1938, V. 19, P. 709–12; abs. in *J. T. I.*, Dec. 1938, P. A731.

A critical study is made of modern drafting systems, including the Jannink, Johannsen, v. Trümbach, Casablancas, Le Blan-Roth, Toenniessen, Kluftinger and Braunmüller systems, with special reference to the provisions for guiding and controlling the fibres, and the advantages and disadvantages of the various systems are pointed out. (C)

HORTOL S: USE IN RAYON SIZING. J. Walther. *Kunstseide*, 1938, V. 20, P. 428–31; abs. in *J. T. I.*, March 1939, P. A160. (C)

LARGE-PACKAGE SPINNING. John Earnshaw. *Text. Wld.*, Feb. 1939, P. 56.

In the writer's opinion the large-package worsted ring frame measures up to the first of the following four requirements, and gives beneficial results on all sides when considering the last question: Can large-package spindles be run at speed limits, either high or low, which will permit efficient and economical production on all counts spun? Is the spun yarn as satisfactory as that spun on other types of equipment? Can large-package spinning frames be used to spin a large variety of counts and qualities? Are such frames as good spinners as other types of equipment? What are the effects, if any, on previous or subsequent processes in manufacture? (C)

RAYON CRÊPE RESEARCH. VI. DETERMINATION OF SOME OF THE VARIATIONS IN NORMAL FIRST QUALITY VISCOSE RAYON. *Am. Dye. Rptr.*, April 3, 1939, P. 163–92.

The Rayon Crêpe Research was conducted jointly by the Throwsters Research Institute, Inc., and the A. A. T. C. C. This part of the Rayon Crêpe Research Report shows the results of an investigation of the physical, optical and dyeing properties of normal deliveries of rayon yarn. The effect of variations found in physical characteristics of rayon yarn and variations in atmospheric humidity during processing upon the pebble of finished crêpe fabrics will be reported upon at a later date. (C)

WOOL FELTING: RESEARCHES ON. J. Schofield. Part I. *J. T. I.*, Oct. 1938, P. T239–T252.

The author contends results of research into the theory of wool felting conducted on woven fabrics are misleading and unreliable, because they are marked by the effect of complex spinning and weaving factors. He notes that a major proportion of the felting research to date has been on cloth, and that "area shrinkage," which is used for evaluation of milling, is not a measure of felting. In his experimental work on "true or natural felt-

ing'' the author uses a felt hardening machine in which the fibre mass is subjected to compression and heat, and the trough and lid to slight opposite oscillations. The only measurable shrinkage of the mass is in thickness, and, for evaluation of felting, porosity measurements of the mass are taken. S. states that results to the date of his writing ''suggest that we are now approaching a stage when a complete statement of the laws of fulling may be made, and that it will be possible to attach an index number to a sample of wool to denote its felting property.'' The author also discusses the subject of ''temperature and wool felting'' both in connection with true felting and cloth milling. His data indicate that felting is promoted by heat up to a temperature of 98° C., this being contrary to previous conclusions of J. B. Speakman and of W. Harrison who found that felting had an optimum temperature of about 45 to 50° C. Perhaps the most interesting and informative result of this paper has been the discussion that it has stimulated between the author, Speakman and Harrison and published in the November and December, 1938, and the January, 1939, issues of *J. T. I.* It not only indicates that the theory and laws of felting are yet to be developed, but basic to their discovery is a scientific definition of felting. Until there is an accepted scientific definition of felting, no scientific method of measuring it can be devised. (C)

III. Chemical and Other Processing (Not Otherwise Classified)

Protein, Starch and Synthetic Products: Use in the Textile Industry. *Klepzig's Textil-Z.*, 1938, V. 41, P. 637–38, 647–48, 658–59; abs. in *J. T. I.*, March 1939, P. A172.

A general discussion of German sources of glues, gelatin, fish protein, casein, starch, products derived from mosses and seaweeds, cellulose derivatives, synthetic resins and polymerized products, and the uses of these materials in the textile industry, especially in sizing and finishing processes. (C)

Rayon: Direct Dyeing. W. Weltzien and K. Windeck-Schulze. *Angew. Chemie*, 1938, V. 51, P. 729–36; abs. in *J. T. I.*, Jan. 1939, P. A33. (C)

Regenerated Cellulose Fibres: Dyeing Properties and Structure. W. Schramek and J. Helm. *Leipz. Monats. Text. Ind.*, 1938, V. 53, P. 309–11; abs. in *J. T. I.*, March 1939, P. A171.

Differences in shade between the outer portion and central portion of a cross-section of a regenerated cellulose fibre when dyed with Congo Rubine, Victoria blue B and Cotton pure blue R are discussed in relation to fibre structure and it is pointed out that dichroism may be due to (1) polydispersity of the colloidal solution of the dye, (2) different arrangement of the adsorbing cellulose surfaces to the optical axis of the microscope, with uniform manner of adsorption of the dye on the cellulose aggregates, and (3) different kinds of dye adsorption on the cellulose, i.e., ordered and irregular adsorption. Of these the third is regarded as most probable and is confirmed by observations of the crystallization of Congo Rubine in the outer layer of the fibre and by the behavior of basic dyes towards cellulose

fibres. Differences in the behavior of basic and substantive dyes are discussed. (C)

SHIRLAN: APPLICATION. Imperial Chemical Industries Ltd. *Text. Rev.*, 1938, V. 6, P. 469–71.

A trade circular is reproduced that gives particulars of the amounts of Shirlan recommended for sizing warps, for addition to Alizarin Purple prints, for treating tent cloth (Waxol being added as well to repel water), for protecting made-up articles, such as leather goods, tents and sails (applied in organic solvent), and for adding to conditioning water for cotton or wool yarns. (C)

SOLUBLE STARCHES: PREPARATION. G. Laqueuille, *Tiba*, 1938, V. 16, P. 677–83; abs. in *J. T. I.*, March 1939, P. A173.

A general account is given of methods for the preparation of soluble starches depending on the use of dilute acids. (C)

STAPLE FIBRE FABRICS: DYEING AND PRINTING. F. Streng. *Mell. Textilber.*, 1938, V. 19, P. 912–15.

A discussion of methods of desizing, washing, bleaching, dyeing, printing and finishing staple fibre fabrics and staple fibre-cotton mixtures, of the materials and apparatus used, and of various difficulties and the means by which they may be overcome. (C)

STAPLE FIBRE, NITROGENOUS STAPLE FIBRE AND LANITAL: DYEING. *Bull. Trim. Lab. Anal. Recherches Ind., Roubaix*, 1938, No. 38, P. 9–13; No. 39, P. 9–14; abs. in *J. T. I.*, Jan. 1939, P. A33. (C)

SULPHUR DYE LIQUORS: ELECTROMETRIC TITRATION. L. I. Belenki, I. I. Ssokolow and M. Je Kasanskaja. *Chloptschtobum. Prom.*, 1937, V. 7, No. 10, P. 32–9 (through *Chem. Zentr.*, 1938, ii, P. 771–72); abs. in *J. T. I.*, Nov. 1938, P. A671. (C)

SURFACE ACTIVE AGENTS IN DYEING: EFFECT OF. J. Edward Smith. *Am. Dye. Rptr.*, Mar. 20, 1939, P. 146–55.

Presented at meeting, New York Section, A. A. T. C. C., Nov. 25, 1938. (C)

TEXTILE FIBRES: "HYDROPHOBING." Velanized Materials: Testing. A. Chwala. *Mell. Textilber.*, 1938, V. 19, P. 905–10.

"Hydrophobing" of fibres is defined as chemical modification of the fibre substance in such a way as to reduce its swelling power and impart water-repelling properties which are resistant to washing and soap and to increase its wet strength. This is effected by blocking the OH-groups in cellulose fibres and the NH_2-groups in wool. "Hydrophobing" processes should be differentiated from impregnating or proofing processes in which a water-repelling effect is produced by mechanical blocking of the spaces in the material, the effect in this case being not generally resistant to washing. Patent processes are classified into (1) "hydrophobing" by (*a*) esterification and (*b*) etherification or formation of acetals and N-contain-

ing analogues, and (2) impregnating processes, and are reviewed. Class (1*b*) includes processes depending on the use of Velan PF. The results of tests of the influence of Velan PF on the moisture absorption and wet strength of rayon and of cotton-staple fibre yarns are given. The effect of the treatment on moisture absorption from moist air increased with concentration of the Velan PF solution used and an appreciable protection against moisture absorption was produced by treatment with a 1% Valan PF bath. The treatment reduced the loss in strength as a result of moisture absorption in the case of the cotton-staple fibre mixture yarn. Increases in actual wet strength as a result of the Velan treatment were not observed. Investigations of the nature of the binding of Velan PF on cellulose have shown that the product of the treatment is the octadecyloxymethyl cellulose ether. "Hydrophobing" treatments giving the desired increase in wet strength have not yet been developed. (C)

TRISODIUM PHOSPHATE: USE IN THE TEXTILE INDUSTRY. P. Colomb. *Teintex*, 1938, V. 3, P. 589–604; abs. in *J. T. I.*, Jan. 1939, P. A30. (C)

UNION DYED FABRICS: NOTES ON FASTENING TO WATER OF. E. S. Chapin. *Am. Dye. Rptr.*, Oct. 31, 1938, P. 610–2.

Paper presented before Northern New England Section, A. A. T. C. C., May 6, 1938. (C)

VAT DYES: APPLICATION. H. Ellner. *Mell. Textilber.*, 1938, V. 19, P. 508–11.

The ideal state for dyeing of textile materials and the ideal state for application of vat dyes are discussed and it is shown that in practice conditions differ considerably from these ideals. Measures adopted by the dyer in order to improve penetration and levelness when dyeing with vat dyes are studied and the influence of the size and shape and nature of the winding of yarn packages on dyeing results is discussed. The importance of satisfactory preliminary treatment, especially thorough desizing and scouring, of fabrics is emphasized. Attempts to improve penetration and levelness by the use of various assistants, the pigment padding process, high temperatures, etc., are critically discussed and a dyeing procedure giving good results with yarns containing staple fibre, in which all the caustic soda and hydrosulphite required are not added at the beginning but are added in suitable proportions to the liquor withdrawn from the goods at intervals, is described. (C)

VISCOSE FILAMENTS: EPIDERMAL STRUCTURE AND DIRECT DYEING. W. Schramek and J. Helm. *Kolloid Z.*, 1938, V. 85, P. 291–94; abs. in *J. T. I.*, March 1939, P. A186.

Viscose filaments were cast without stretching, and cross-sections were prepared and stained with Bordeaux extra, Sirius BL, Malachite green, Cotton blue 4R, and Kongorubin. A colored plate shows the appearance of the sections as in the dye and after rinsing with water. Clear evidence of a "skin" is given and this is discussed in relation to theories of direct dyeing. (C)

VISCOSE RAYON: CLASSIFIED LIST OF DIRECT DYES FOR USE WITH. J. Boulton. Communication in *J. Soc. Dyers Col.*, June 1938, P. 268–74. (C)

WEIGHTED SILK: PROTECTION AGAINST TENDERING BY LIGHT. A. Weilenmann. *Bull. Fed. Inter. Assoc. Chim. Text. et Couleur*, 1938, V. 3, P. 157–80; abs. in *J. T. I.*, Dec. 1938, P. A749. (C)

WETTING AGENTS IN TEXTILE PROCESSING. IV. DERIVATIVES OF ARYLAMINE SULPHONIC ACIDS. I. S. Uppal and K. Venkataraman. *J. Soc. Dyers Col.*, March 1939, P. 125–34. (C)

WOOL: ACTION OF HYPOCHLOROUS ACID ON. H. J. Henk. *Mell. Textilber.*, 1938, V. 19, P. 989–90.

The mode of action of hypochlorous acid on wool is influenced by pH. In acid solution, chlorine is the active agent; in neutral solution, hypochlorous acid predominates and the reaction is primarily one of oxidation; in alkaline solution, addition of hypochlorous acid to unsaturated linkages occurs. Oxidation and addition reactions are deleterious, and treatment at pH 4.9–5.1 favors the desirable chlorinating reaction. It is suggested that no undesirable secondary reactions occur when sulphuryl chloride is used. (C)

WOOL: ACTION OF LIGHT ON. *Wool Record & Text. Wld.*, Sept. 15, 1938, P. 609–11.

The subject is discussed with reference to the accelerating and retarding effect of certain dyes and chemicals. (C)

WOOL: EFFECT OF ARSENIC AND COPPER SULPHATE ON DYEING QUALITIES OF. *Agric. Gazette of New South Wales*, 1938, V. 49, P. 602–3; abs. in *J. T. I.*, Feb. 1939, P. A108.

It is concluded that there is no reason to consider that dipping in a suitable mixture containing arsenic would have any deleterious effect on subsequent dyeing processes, but that copper sulphate may very definitely interfere with results obtained normally. (C)

WOOL: UNSHRINKABLITY. K. H. Fischer. *Deut. Textilwirt.*, 1938, V. 5, No. 20, P. 6–9; abs. in *J. T. I.*, Feb. 1939, P. A109.

Hall's sulphuryl chloride process is criticized and compared with the German Hypak process (see *J. T. I.*, 1937, P. A331 and A702). (C)

WOOL: REACTIVITY OF THE SULPHUR LINKAGE IN. J. B. Speakman, B. Nilssen and G. H. Elliott. *Nature*, 1938, V. 142, P. 1035.

The unshrinkable finish imparted to wool by various processes depends on the formation of a gelatinous degradation product of keratin, on or under the scale structure of the fibres. This may be accomplished by means of any oxidizing reagent (such as chlorine) which is capable of breaking down the disulphide bonds. Sulphuryl chloride acts in a similar manner, since it reacts with dibenzyl disulphide at 37° C. to give dibenzyl disulphoxide, benzyl sulphonyl chloride and benzyl chloride. (C)

IV. Research Methods and Apparatus

Cotton: Volumetric Estimation of Moisture with Particular Reference to. N. C. Mitra and K. Venkataraman. *J. S. C. I.*, 1938, V. 57, P. 306–10; abs. in *J. Soc. Dyers Col.*, March 1939, P. 170.

Moisture in cotton has been previously estimated by oven-drying or distillation with an organic liquid. A new method is described in which the material is refluxed with acetic anhydride in solvent naphtha, the excess acetic anhydride being treated with aniline to form acetanilide and one mol. of acetic acid, the total acetic acid formed being then determined by titration. The method should be applicable to many substances and commercial products for which other methods are unsuitable. (C)

Dispersions of Cellulose in Cuprammonium Solution: Measurement of Apparent Fluidity of. Ralph T. Mease. *Ntl. B. S. J. Rsch.*, March 1939, P. 271–84; RP1179.

This paper describes relatively simple equipment and technique found practical for measurement of the apparent fluidity of dispersions of cellulose in cuprammonium solution. Directions are given for the preparation and storage of the cuprammonium solution; for the construction and calibration of viscometers; for the dispersion of cellulose in the cuprammonium solution; and for the measurement of the fluidity of the dispersion. Some of the difficulties which arise in precise measurements are indicated, and data illustrating the duplicability of results are presented. (C)

Egyptian Cotton 1935–36 Crop: Spinning Tests. H. A. Hancock. *Min. Agric. Egypt, Tech. Sci. Serv., Bot. Sec., Bull.* No. 189, 58 pp. (1938).

An account is given of the technique employed at the Spinning Test Mill, Giza. The standard error of a spinning test is shown to be about 1%. In comparing the spinning values of old or new varieties, the strength level to which the yarns are spun is found to be of little consequence. Providing the cottons are given identical treatment, they rank in much the same order whether spun from combed or from carded preparation, in fine or in coarse counts, in warp twist or in doubling weft, over the range of Egyptian cottons. A spinning in a medium counts carded yarn gives sufficiently accurate information for most purposes. A report on the yarn strength of mixing shows that the strength of any 50: 50 mixing is near to the average strength of the components. The predominant influence of hair clinging power whatever the twist, is emphasized. The strength at low twist appears to be the basis on which corrections should be made, by factors at present unknown, in order to predict the strength at higher twists. An experiment in varying the feed of cotton to the carding engine showed that fast thin laps produced stronger yarns than slow thick ones. Parallel-haired laps produced, on the other hand, inferior results to normal laps. (C)

Fabrics: Bending Resistance; Use for Evaluation of Effects of Textile Chemical Treatments. F. Kurtz. *Klepzig's Textil-Z.*, 1938, V. 41, P. 593–603; abs. in *J. T. I.*, Feb. 1939, P. A125.

The determination of the bending resistance of textile materials by the Schopper method is discussed and the use of a quantity termed the

bending resistance value KG is recommended as a basis for determining the effects of various treatments on the materials. This bending resistance value KG is given by the relation $0.5\ a + 0.3\ b + 0.2\ c$, where a, b and c are the numbers of bends producing break under loads of 10% 20%, and 30% of the tensile strength, respectively. Tables and graphs showing the effects of treating woolen yarns with soda solution and of acid dyeing treatments, the effects of scouring and bleaching cotton fabrics, the effects of dyeing staple fibre-cotton fabrics with Indanthrene dyes, and the effects of treatment of viscose rayon fabrics with various finishing agents on the strength, bending resistance and other physical properties of the textile materials are given and discussed. The usefulness of the bending resistance value as a measure of the effects of such chemical treatments is emphasized. (C)

FIBRES: COMPARISON MICROSCOPY. A. Herzog. *Kunstseide*, 1938, V. 20, P. 382–89; abs. in *J. T. I.*, Feb. 1939, P. A124.

The value of photo-micrographs for comparison purposes is pointed out but the use of comparison microscopes for the direct preparation of photomicrographs is not recommended. (C)

FABRICS: MEASUREMENT OF WEAR AND SLIPPING RESISTANCE. W. Weltzien. *Monatsh. Seide u. Kunstseide*, 1938, V. 43, P. 334–48; abs. in *J. T. I.*, Dec. 1938, P. A765. (C)

FIBRES: DARK-FIELD MICROSCOPY. P. A. Koch. *Klepzig's Textil Z.*, 1938, V. 41, P. 138–42, 226–32, 316–27, 441–44, 491–98; abs. in *J. T. I.*, Dec. 1938, P. A760.

The author reviews the results of earlier investigations of fibres by means of the ultra-microscope and the various theories of the fine structure of natural and artificial fibres and then gives an account of improved apparatus for dark-field microscopy and the results of studies of plant, animal, mineral and artificial fibres, including mercerized, bleached, dyed delustred, animalized and other modified fibres. Observations of the swelling of plant fibres, rayon and staple fibre in cuprammonium and calcium thiocyanate solutions are also described. Numerous photomicrographs are given. (C)

V. PURE SCIENCE, ECONOMICS AND MISC.

FLUORESCENCE AND PHOSPHORESCENCE. J. H. Gisolf and W. de Groot. *Philips Tech. Rev.*, Aug. 1938, V. 3, P. 241–47 (See *Ibid*, 1938, V. 3, P. 125); abs. in *Eastman Kodak Abs. Bull.*, Jan. 1939, P. 38.

The phenomena of fluorescence and phosphorescence of a series of different compounds are discussed. Tentative explanations regarding the mechanism of luminous emission in different cases are advanced on the basis of investigations of the afterglow. (S)

FOAM IN GELATIN. D. Virnik and F. Blyumin. *Kino Photo Chem. Ind.*, 1938, No. 4, P. 37–40; abs. in *Eastman Kodak Abs. Bull.*, Jan. 1939, P. 48.

The effect of mixtures of sulfonaphthenic acid and sulfite on the formation and the stability of foam in gelatin solutions was studied. Comments

are made on the theory of foam formation, and the methods of measuring foaming are described. The decrease of the stability of foams during the process of coating gelatin solutions is believed to be due to temperature changes. Foam is more easily obtained and is more stable for filtered than for unfiltered gelatin. (S)

FOAMS: MECHANICAL PROPERTIES. A. Siehr. *Kolloid-Z.*, 1938, V. 85, P. 70–4; abs. in *J. T. I.*, Feb. 1939, P. A135.

Experiments on protein foam, produced from the diluted, expressed juice of potatoes showed that the foam acts as a viscous liquid when passing through tubes and like a gas under compression or expansion. Because of the high viscosity and compressibility, the foam displays the specific property of "pressure accumulation" on passage through narrow tubes. Suggestions are given for avoiding the disturbances caused by this accumulated pressure in foam-producing apparatus. (C)

GELATIN AND ANIMAL GLUES: PREPARATION AND PROPERTIES. A. Lambrette. *Tiba*, 1938, V. 16, P. 421–33; abs. in *J. T. I.*, Dec. 1938, P. A779.

A general account is given of the nature, preparation and properties of gelatin and animal glues, and of methods of analyzing and testing these products especially in relation to their suitability for use in textile finishing processes. (C)

HEAT OF REACTION OF WOOL WITH ACIDS. J. B. Speakman and E. Stott. *Trans. Faraday Soc.*, 1938, V. 34, P. 1203–9.

Determination of the heat of reaction of wool with acid solutions of varying pH, indicates that at low pH values, the heat of reaction per gram equivalent of wool (1250 g.) is greater in monochloroacetic acid than in hydrochloric acid. This result, which is clearly related to the greater swelling and acid-combining capacity of wool in concentrated solutions of the weaker acid, has been referred to the liberation of — NH — groups for combination with acid when the sheets of covalently likened peptide chains which form the micelles of the fibre are separated. In keeping with this deduction is the fact that the heat of reaction per gram-molecule of combined acid decreases with fall of pH below 2.0 in the case of monochloroacetic acid. With hydrochloric acid, on the other hand, the heat of reaction per gram-molecule of combined acid is independent of pH below 2.0, the average value being 3.56 k.cal. This value is so low as to indicate that the basic side chains of wool are not free, but are combined with acid side-chains to form salt linkages, as has been previously suggested. (C)

KERATIN: STRUCTURE OF. B. Nilssen. *Tids. Kjemi Bergvesen*, 1938, V. 18, P. 2–7 (through *C. A.*, 1938, V. 32, Col. 9507); abs. in *J. T. I.*, Feb. 1939, P. A142.

A review of the literature of the structure of keratin, and the mechanical properties of wool. (C)

MICELLAR THEORY AS ILLUSTRATED BY THE FINE STRUCTURE OF FIBRES. A. Frey-Wyssling. *Kolloid-Z.*, 1938, V. 85, P. 148–58; abs. in *J. Soc. Dyers Col.*, March 1939, P. 163.

A discussion of the polarized light and X-ray methods of examining the fine structure of cellulosic fibres indicates that the reticular system will

play a highly important part in the future examination of biological fine structure, and, therefore, a terminology adapted for use in a biological sense is essential. Thus, the gel framework may be defined as the micellar portion, and the pores, imbibition liquid or incrustation substance, as the intermicellar portion of the fibre. Reactions between the framework beams are described as intermicellar and those in the beams as intramicellar. Gels with a micellar framework are distinguished from those possessing a molecular framework in that chemical and mechanical processes, *e.g.*, solution, viscosimetry, or double refractivity, are necessary to examine the morphology of the highly polymeric molecular framework, while the fine structure of the micellar framework can be explained entirely by means of polarization or X-ray examination without reference to the internal structure. (C)

Photoluminescent Properties of Synthetic Flavine. C. Dhéré and V. Castelli. *Comp. rend.*, June 27, 1938, V. 206, No. 26, P. 2003–05; abs. in *Eastman Kodak Abs. Bull.*, Jan. 1939, P. 46. (S)

Book Reviews

Enforcement of Virgin Wool Legislation. National Association of Wool Manufacturers, New York, N. Y. 20 pp.

In a pamphlet of 20 pages is presented reports of four laboratories demonstrating conclusively as a result of scientific tests that there is no chemical or physical analysis that can indicate the content of new wool and reprocessed wool in a fabric, and that lacking a laboratory test there can be no enforcement of legislation which compels such distinction. The laboratory findings were based upon analyses of a group of fabrics manufactured specially for these tests, the components of which fabrics were certified but were unknown to the laboratories. One of the laboratories conducted a series of tests designed to show "fabric merit" of each fabric based upon the following physical characteristics: (1) strength-weight ratio, (2) fibre length, (3) percentage of frayed and broken ends. The results when compared with the actual new wool content, as reported by the manufacturers of each fabric, showed a complete lack of relationship between "fabric merit" and new wool content. The tabulation of these laboratory findings are given in a leaflet entitled "The Irrelevancy of 'Virgin' Wool Content to Fabric Quality." (C)

Microscopic Methods Used in Identifying Commercial Fibres. Thora M. Plitt. Circular of the National Bureau of Standards C423, 26 pp., for sale by the superintendent of Documents, Washington, D. C., price 10c.

A compilation of about 50 microscopic methods for the identification of paper, textile, and cordage fibres has been made. During a survey of the literature these were selected and tried in the fibre-testing laboratory. Their relative merits are discussed. In addition to inspection by the aid of magnification, the means of approach include staining reactions, ashing preparations, and the use of polarized and ultraviolet light. References to original sources and standard pertinent works are appended. (C)